

Degree of Conversion and Polymerization Shrinkage of Bulk-Fill Resin-Based Composites

P Yu • AUJ Yap • XY Wang

Clinical Relevance

Bulk-fill resin-based composites (RBCs) are not a homogeneous group of materials. Their degree of conversion and polymerization shrinkage are product/depth dependent. Bulk-fill RBCs should generally not be cured in more than 4-mm increments. As the degree of conversion of some bulk-fill products is low, they should be used with caution clinically.

SUMMARY

This study evaluated the degree of conversion (DC) and polymerization shrinkage (PS) of contemporary bulk-fill resin-based composites (RBCs) including giomer materials. Two giomer bulk-fill (Beautiful Bulk Restorative [BBR], Beautiful Bulk Flowable [BBF]), two nongiommer bulk-fill (Tetric N-Ceram Bulk-fill [TNB], Smart Dentin Replacement [SDR]), and three conventional non-bulk-fill (Beautiful II [BT], Beautiful Flow Plus [BF], Tetric N-Ceram [TN]) RBCs were selected for the study. To evaluate the DC, disc-shaped specimens of 5-mm diameter and 2-mm, 4-mm, and 6-mm thickness were

fabricated using customized Teflon molds. The molds were bulk filled with the various RBCs and cured for 20 seconds using a light-emitting diode curing light with an irradiance of 950 mW/cm². The DC (n=3) was determined by attenuated total reflectance Fourier transform infrared spectroscopy by computing the spectra of cured and uncured specimens. PS (n=3) was measured with the Acuvol volumetric shrinkage analyzer by calculating specimen volumes before and after light curing. The mean DC for the various materials ranged from 46.03% to 69.86%, 45.94% to 69.38%, and 30.65% to 67.85% for 2 mm, 4 mm, and 6 mm, respectively. For all depths, SDR had the highest DC. While no significant difference in DC was observed between depths of 2 mm and 4 mm for the bulk-fill RBCs, DC at 2 mm was significantly greater than 6 mm. For the conventional RBCs, DC at 2 mm was significantly higher than at 4 mm and 6 mm. Mean PS ranged from 1.48% to 4.26% for BBR and BF, respectively. The DC at 2 mm and PS of bulk-fill RBCs were lower than their conventional counterparts. At 4 mm, the DC of giomer bulk-fill RBCs was lower than that of nongiommer bulk-fill materials.

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INTRODUCTION

Chemically cured resin-based composites (RBCs) were first introduced as a replacement for silicate cements and autopolymerizing acrylic materials. Although they are esthetically superior, their poor clinical performance did not permit their use in posterior teeth.¹ With developments in dental RBC/adhesive technologies and trends toward more conservative cavity preparations, light-cured RBCs are now routinely used to restore posterior teeth and account for approximately half of all posterior direct restorations placed.² Posterior RBC restorations are, however, technically challenging and time consuming to perform as they require incremental 2 mm material placements and light curing due to depth of cure and polymerization shrinkage (PS) issues. Inadequately cured RBCs have reduced physicochemical qualities and chemical stability and are potentially toxic to pulpal tissues.^{3,4} Cited deleterious effects of polymerization-induced shrinkage stress include marginal leakage, gap formation, cuspal deflection, tooth cracking, and reduced mechanical properties of RBC restorations.⁵ In addition to increased clinical time and technical complexities, other disadvantages of the incremental filling technique include reduced bond strengths as well as voids, contamination, and bond failures between adjacent RBC layers.⁶⁻⁹

Innovative bulk-fill RBCs with claims of enhanced depth of cure and reduced PS have been introduced to the dental profession. These materials can apparently be placed in 4-mm increments, reducing the time and effort required for layering and adapting posterior RBCs. Strategies used to achieve bulk filling included the use of novel proprietary resins, special modulators, unique fillers, and filler control. At such increments, light transmission through RBCs may be compromised, leading to reduced monomer to polymer conversion.¹⁰ The depth of cure and degree of conversion (DC) of bulk-fill RBCs have been investigated using a variety of methods. These include the ISO scraping test,¹¹ microhardness test, Fourier transform infrared (FTIR), and Raman spectroscopy. While some studies have conveyed adequate cure and DC at a depth of 4 mm,¹²⁻¹⁵ others have reported contradictory outcomes.^{16,17} The variances can be attributed to disparities in testing methodologies as well as materials evaluated. Bulk-fill RBCs are not a homogeneous group of materials, and differences in chemistry, viscosities, filler type, and quantity exist. PS studies showed that bulk-fill RBCs generally

have lower shrinkage than conventional materials.¹⁷⁻¹⁹

DC and PS data on recently launched giomer bulk-fill materials are, however, scarce.^{20,21} Giomers are based on prereacted glass ionomer (PRG) filler technology and are also known as PRG-RBCs. PRG fillers are obtained by reacting fluoride-containing glass with polyacids in the presence of water. The resultant product is freeze-dried, milled, silanized, and ground prior to being incorporated into silica-filled resins. Giomer RBCs are capable of fluoride release/recharge²² and possess antibacterial properties.^{23,24} Clinical qualities of most posterior giomer restorations were found to be acceptable even after 13 years of service.²⁵ Use of PRG fillers in giomer bulk-fill restoratives may, however, affect both DC and PS. The null hypotheses were as follows: 1) no significant differences exist in DC and PS between contemporary bulk-fill RBCs including giomer materials and 2) cavity depths do not influence DC of the various bulk-fill and conventional RBCs.

METHODS AND MATERIALS

The materials selected for the study included two giomer bulk-fill (Beautifil Bulk Restorative [BBR], Beautifil Bulk Flowable [BBF]), two nongiomer bulk-fill (Tetric N-Ceram Bulk-fill [TNB], Smart Dentin Replacement [SDR]), and three conventional non-bulk-fill (Beautifil II [BT], Beautifil Flow Plus [BF], Tetric N-Ceram [TN]) RBCs. The technical profiles of the materials are reflected in Table 1.

Customized Teflon molds with cylindrical recesses of 5-mm diameter and 2-mm, 4-mm, and 6-mm depths were fabricated. A transparent polyester strip (Striproll, KerrHawe, Bioggio, Switzerland) was placed at the bottom of the molds. The molds were then bulk filled with the various RBCs in a single increment, and excess material was extruded by application of pressure through a glass slide. The materials were then light polymerized from the top surface using a light-emitting diode (LED) curing light (BluePhase, Ivoclar Vivadent, Shaan, Liechtenstein) with a curing tip diameter of 8 mm and an irradiance of 950 mW/cm² for 20 seconds. The irradiance of the curing light was assessed with an LED light tester (FB-M2000A, Fibop Medical Instrument, Foshan, China). Disc-shaped specimens (n=3) of 5 mm diameter and 2-mm, 4-mm, and 6-mm thickness were obtained. The DC of the bottom surfaces of the specimens was measured immediately after light polymerization using an FTIR spectrometer (Tensor 27, Bruker Optics, Ettlingen, Germany) with an attenuated total reflectance

Table 1: *Technical Profiles of the RBCs Evaluated in the Study*

Materials	Abbreviation	Type	Composition		Filler Content (wt%/vol%)	Filler size (μm)	Shade	Manufacturer	Lot No.
			Resin	Filler					
Beautifil Bulk Restorative	BBR	Bulk-fill restoratives	Bis-GMA, UDMA, Bis-MPEPP, TEGDMA	S-PRG filler based on fluoroboroaluminosilicate glass	87.0/74.5	N.A	U	Shofu Inc, Kyoto, Japan	011510
Beautifill	BT	Conventional restoratives	Bis-GMA, TEGDMA	Multifunctional glass filler, S-PRG filler based on fluoroboroaluminosilicate glass	83.3/68.6	0.01-4, mean 0.8	A1	Shofu Inc, Kyoto, Japan	041460
Beautifil Bulk Flowable	BBF	Bulk-fill flowables	Bis-GMA, UDMA, Bis-MPEPP, TEGDMA	S-PRG filler based on fluoroboroaluminosilicate glass	72.5/NA	NA	U	Shofu Inc, Kyoto, Japan	011506
Beautifil Flow Plus F00	BF	Conventional flowables	Bis-GMA, TEGDMA	Multifunctional glass filler, S-PRG filler based on fluoroboroaluminosilicate glass	67.3/47.0	0.01-4, mean 0.8	A1	Shofu Inc, Kyoto, Japan	011530
Tetric N-Ceram Bulk Fill	TNB	Bulk-fill restoratives	Dimethacrylates	Barium glass, ytterbium trifluoride, mixed oxide, and copolymers	80-81/55-57	0.04-3	A1	Ivoclar Vivadent AG, Schaan, Liechtenstein	S24104
Tetric N-Ceram	TN	Conventional restoratives	Dimethacrylates	Barium glass, prepolymer, ytterbium trifluoride, and mixed oxide	75-77/53-55	0.04-3, mean 0.6	IVA	Ivoclar Vivadent AG, Schaan, Liechtenstein	T29061
Smart Dentin Replacement	SDR	Bulk-fill flowables	Modified UDMA, EBPADMA, TEGDMA	Barium-alumino-fluoro-borosilicate glass, strontium alumino-fluoro-silicate glass	68/45	Mean 4.2	U	Dentsply Caulk, Milford, DE, USA	1410000524

Abbreviation: Bis-GMA, bisphenol A glycidyl dimethacrylate; Bis-MPEPP, 2,2-bis[(4-methacryloxy polyethoxy)phenyl]propane; EBPADMA, ethoxylated bisphenol A dimethacrylate; NA, not available; S-PRG: surface prereacted glass ionomer; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.

(ATR) accessory (MIRacle, PIKE Technologies, Madison, WI, USA). The ATR crystal was placed in close contact with the bottom surface, and FTIR spectra ranging from 600 to 4000 cm^{-1} were documented by 32 scans at a resolution of 4 cm^{-1} . The FTIR spectra of uncured RBCs were recorded at the start of the experiment, and DC was calculated using the following formula:

$$\text{DC}\% = \left[1 - \frac{\text{Cured} (1638\text{cm}^{-1}/\text{internal standard})}{\text{Uncured} (1638\text{cm}^{-1}/\text{internal standard})} \right] \times 100$$

The peak height around 1638 cm^{-1} , indicating the absorbance intensities of aliphatic C=C, was calibrated according to Rueggeberg and others.²⁶ The Peak of aromatic C=C around 1608 cm^{-1} (1600 cm^{-1} for SDR due to the lack of aromatic C=C) was taken as the internal standard and also calibrated.

PS (n=3) was measured by means of a video imaging device (AcuVol volumetric shrinkage analyzer, Bisco Inc, Schaumburg, IL, USA) in volumetric reconstruction mode. The RBC specimens were manually shaped into a hemisphere and placed on the rotational polytetrafluoroethylene pedestal inside the AcuVol chamber in front of the CCD camera. The specimens were imaged at a distance of 10 cm and subsequently irradiated for 20 seconds using the same LED curing light as for DC. The images were digitized and analyzed with the proprietary image-processing software. The volume of the specimens before and after curing was recorded as V_1 and V_2 , respectively. The PS for the various RBCs was calculated as follows:

$$\text{PS}\% = \left[(V_1 - V_2) / V_1 \right] \times 100$$

Statistical analysis was carried out using SPSS version 20.0 (IBM SPSS Inc, Chicago, IL, USA). DC

Table 2: Mean Degree of Conversion (Standard Deviations) for the Various RBCs at the Various Depths^a

Material	2 mm	4 mm	6 mm
BBR	46.03 (0.66) ^{Aa}	45.94 (0.66) ^{Aa}	41.26(0.19) ^{Ba}
BT	62.78 (0.43) ^{Abc}	56.38 (0.14) ^{Bb}	35.24(0.79) ^{Cb}
BBF	55.53 (2.03) ^{Adf}	53.17 (0.81) ^{ABbc}	49.04(3.20) ^{Bc}
BF	66.11 (3.49) ^{Abe}	50.80 (2.05) ^{Bc}	30.65(0.56) ^{Cd}
TNB	56.22 (0.24) ^{Adf}	55.46 (2.02) ^{Ab}	49.02(1.35) ^{Bc}
TN	59.78 (0.17) ^{Acf}	56.11 (0.32) ^{Bb}	38.51(0.73) ^{Cab}
SDR	69.86 (0.41) ^{Ae}	69.38 (0.14) ^{Ad}	67.85(0.93) ^{Be}

^a Different uppercase letters in each row and different lowercase letters in each column indicate significant differences within the same materials and in the same increments, respectively ($p < 0.05$; Tukey HSD test).

and PS data were subjected to one-way analysis of variance and Tukey HSD test at a significance level of 0.05. Differences in DC and PS between RBCs as well as differences in DC between depths of 2 mm, 4 mm, and 6 mm for each material were examined. The relationship between DC at 2 mm and PS was also analyzed with Pearson’s correlation at a significance level of 0.05.

RESULTS

Mean DC and standard deviations of the various RBCs at the different depths are reflected in Table 2. Table 3 shows the DC obtained at 4 mm and 6 mm expressed as a percentage of DC at 2 mm. The latter was used as the reference as it offered the highest DC in our study and was the recommended depth of cure for most conventional RBCs. Mean PS and standard deviations of the various RBCs are shown in Table 4. Results of statistical analysis of DC and PS are displayed in Tables 2 and 4, respectively.

Mean DC for the various RBCs ranged from 46.03% to 69.86%, 45.94% to 69.38%, and 30.65% to 67.85% for 2 mm, 4 mm, and 6 mm, respectively. For all depths, SDR had the highest DC. For 2 mm and 4 mm depths, DC of BBR was the lowest. BF had the

Table 3: DC at 4 and 6 mm Expressed as a Percentage of DC at 2 mm

Material	2 mm	4 mm	6 mm
BBR	100	99.8	89.6
BT	100	90.0	56.1
BBF	100	95.8	88.3
BF	100	76.8	46.4
TNB	100	98.6	87.2
TN	100	93.9	64.4
SDR	100	99.3	97.1

Table 4: Mean Polymerization Shrinkage (Standard Deviation) for the Various RBCs^a

Material	Volumetric Shrinkage (%)
BBR	1.48 (0.01) ^A
BT	2.21 (0.03) ^B
BBF	3.02 (0.04) ^C
BF	4.26 (0.02) ^D
TNB	2.10 (0.01) ^E
TN	2.11 (0.02) ^E
SDR	3.38 (0.04) ^F

^a Different uppercase letters in each row indicate significant differences within the same materials ($p < 0.05$; Tukey HSD test).

lowest DC values at 6 mm. At 4 mm, the decrease in DC ranged from 0.2% to 4.2% for bulk-fill RBCs and 6.1% to 23.2% for conventional materials. At 6 mm, the reduction in DC ranged from 2.9% to 12.8% and 35.6% to 53.6% for bulk-fill and conventional RBCs, respectively. While no significant difference in DC was observed between depths of 2 mm and 4 mm for the bulk-fill RBCs, DC at 2 mm was significantly greater than 6 mm. For the conventional RBCs, DC at 2 mm was significantly higher than at 4 mm and 6 mm. Significant differences in DC were also observed between 4 mm and 6 mm for the conventional materials.

Differences in DC between RBCs were found to be product and depth dependent. At 2-mm depth, SDR had significantly higher DC than most other RBCs, with the exception of BF. BBR had significantly lower DC when compared with the other RBCs. The DC of giomer bulk-fill RBCs was significantly lower than that of their conventional giomer counterparts. No significant difference in DC was observed between TNB and TN. At 4 mm, SDR had significantly greater while BBR had significantly lower DC than all the other RBCs including BT. No significant difference in DC was observed between BBF and BF nor between TNB and TN. At 6-mm depth, significantly greater DC was observed for SDR when compared with the other materials. DC of the bulk-fill RBCs was higher than their conventional equivalents.

Mean PS ranged from 1.48% to 4.26% (Table 4; Figure 1). Ranking of PS from highest to lowest was as follows: BF > SDR > BBF > BT > TN = TNB > BBR. The PS of BF was significantly greater while that of BBR was significantly lower than all the other RBCs. The PS of the giomer bulk-fill RBCs was significantly lower than that of their conventional giomer counterparts. No significant difference in PS was observed between TNB and TN. A moderate,

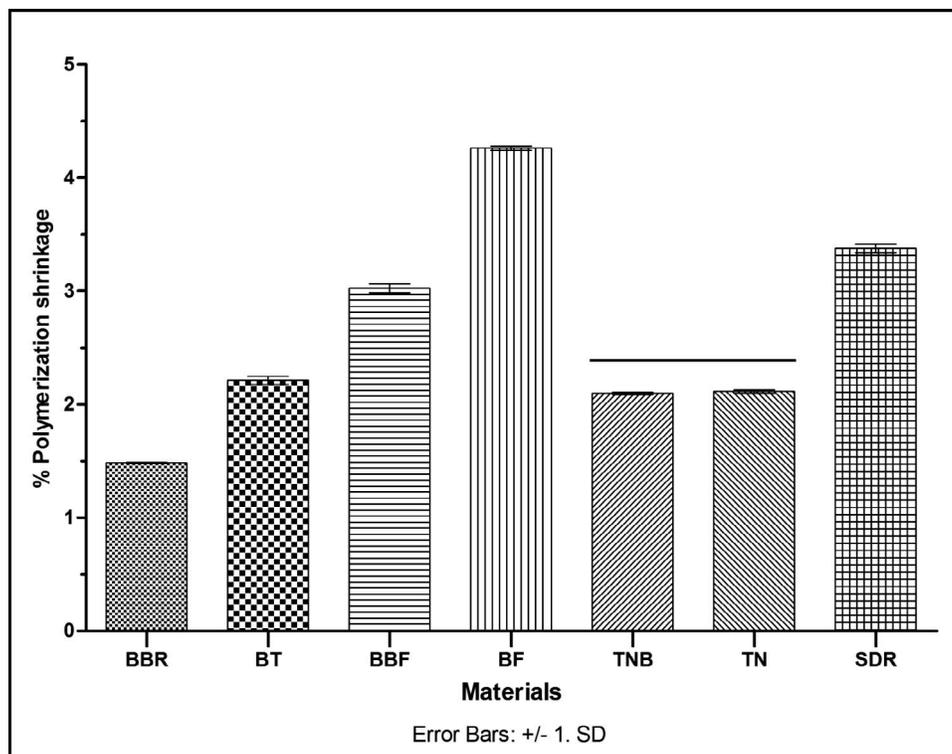


Figure 1. Mean volumetric shrinkage (%) for the various RBCs. Horizontal line indicates homogeneous grouping. Results of Tukey HSD test at a significance level of 0.05.

positive, and significant correlation was observed between DC and PS ($r=0.72$, $r^2=0.52$, $p=0.0002$) with Pearson correlation.

DISCUSSION

We compared the DC and PS of contemporary bulk-fill RBCs including restorative and flowable giomer products. Based on the results of this study, the null hypotheses were rejected. FTIR and Raman spectroscopy were first used to study water sorption in dental resins and were subsequently employed to analyze DC in methacrylate polymerization.^{27,28} Other techniques of measuring DC include electron paramagnetic resonance, nuclear magnetic resonance, differential scanning calorimetry, and differential thermal analysis.²⁹⁻³² FTIR, however, remains the most frequently used technique.³³ DC was determined by the proportion of remaining aliphatic C=C double bonds' concentration in the cured RBCs relative to the total number of C=C bonds in the uncured materials. DC was reported to be significantly influenced by light source and RBC materials.³⁴ Light source variables include type of light, power density, wave length, irradiation time, irradiation distance, and light-activation method, which were all standardized in our study. DC was found to be both product and depth dependent. The percentage decrease in DC with increased depth was RBC

dependent, with bulk-fill materials exhibiting better conversion at increased depths when compared with their conventional counterparts (Table 3). Significant differences in DC were observed between 2 mm, 4 mm, and 6 mm depths for conventional materials but only between 2 mm and 6 mm depths for bulk-fill RBCs. Although results supported manufacturers' claims of bulk filling in 4 mm increments for the bulk-fill RBCs evaluated, the DC of bulk-fill giomer materials was relatively low (Table 2).

The DC of C=C double bond for RBCs generally ranges from 55% to 65%.³⁵ The DC of the bulk-fill restorative giomer BBR was less than 55%, even at the 2 mm depth, while the DC of the bulk-fill flowable giomer BBF was marginally greater than 55% at 2 mm. The DC of conventional giomer RBCs at 2 mm (62.8% for BT and 66.1% for BF) was significantly higher than that of BBF and BBR. The low DC of bulk-fill gomomers (53.2% for BBF and 45.9% for BBR) at 4 mm in this study concurred with the findings of Al-Ahdal and coworkers.²⁰ They measured DC at a 4 mm depth after 20 seconds of light curing with an irradiance of 1200 mW/cm² and reported a DC of 56.3% for BBF and 38.9% for BBR. Ilie and Fleming²¹ conducted a similar study with a higher irradiance of 1415 mW/cm² and also reported similar DC values of 57.7% and 40.0% for BBF and BBR, respectively. DC may not be optimized imme-

diately or 5 minutes after light curing, and some degree of postpolymerization conversion is anticipated for up to 24 hours. Even after 24 hours, the DCs of BBF and BBR were 65.7% and 49.7%, respectively. In comparison, the DC at 24 hours of other bulk-fill RBCs ranged from 54.5% to 71.9%.²⁰ As both BBF and BBR are similar in resin chemistry and filler types, differences in DC can be attributed to variation in filler quantity. BBF, which is flowable, has a lower filler content (72.5 wt%) than BBR (87.0 wt%). DC has been shown to decrease proportionally with increasing filler content and can be attributed to the light scattering at the resin-filler interfaces.³⁶ In addition to filler quantity, filler type, size, and shape can also influence the efficiency of light scattering.³⁷

At all depths, SDR had the highest DC. At 4 mm and 6 mm, the decrease in DC was only 0.7% and 2.9% correspondingly. The high DC of SDR was in accordance with other works³⁸ and can be ascribed to its UDMA-based resin matrix and large filler particles. Sideridou and others³⁹ studied the effect of chemical structure on DC in light-cured dimethacrylate-based dental resins and found DC increased in the order Bis-GMA < Bis-EMA < UDMA < TEGDMA. UDMA has lower viscosity and higher molecular flexibility than BisGMA. It integrates an imino (-NH-) group for chain transfer reactions that offer another path for the continuation of polymerization. The large filler particles (mean 4.2 μm) used in SDR reduces filler-matrix interfaces. Incident light interference and scattering are reduced, increasing translucency, light penetration, and cure.⁴⁰ TNB contains a germanium-based photo-initiator (Ivocerin) that generates at least two free radicals for initiating polymerization initialization. Camphorquinone, the most widely used visible-light photo-initiator in RBCs, generates only one radical. The fore mentioned explains in part the significantly higher DC of TNB when compared with TN at 6 mm. According to the manufacturer, TNB is also more translucent (15%) than TN (10%), allowing for more light penetration and greater depth of cure.¹⁵ The decrease in DC at 4 mm and 6 mm was only 1.4% and 12.8%, respectively, for TNB (Table 3).

PS of RBCs had been determined with a variety of techniques, including water/mercury dilatometry, cuspal deflection, specific gravity analysis, electrical strain gauges, and optical measurements.⁴¹ The optical video-imaging technique was selected as it provided an easy method for measuring volumetric shrinkage. In addition, the AcuVol video-imaging technique had been shown to give reproducible

results for volumetric shrinkage comparable with those measured by dilatometry.⁴² Volumetric shrinkage for conventional methacrylate-based RBCs ranges from 2% to 6%.⁴³ Mean PS for the bulk-fill RBCs evaluated ranged from 1.5% to 3.4%, while that of conventional materials ranged from 2.1% to 4.3%. While no significant difference in PS was observed between TNB and TN, the bulk-fill giomers BBF and BBR had significantly lower shrinkage than their conventional counterparts BF and BT. Our findings corroborated those of studies using other shrinkage measurement techniques that reported generally lower shrinkage with bulk-fill materials.¹⁷⁻¹⁹ PS of the flowable giomers (BBF and BF) were significantly greater than their restorative equivalents (BBR and BT). The difference in PS can be attributed to filler volume fraction and the DC of the RBCs.⁴⁴ Flowable giomers with their lower filler and higher resin content are anticipated to shrink more than their highly filled restorative counterparts. The correlation between DC and PS was moderate, positive, and significant. SDR, which had the highest DC, consequently had the highest shrinkage. The lower PS of bulk-fill giomers is also explained by their lower DC at 2 mm when compared with conventional equals. DC of the conventional giomers BT and BF was 62.8% and 66.1%, while that of the bulk-fill giomers BBR and BBF was only 46.0% and 55.5%. Further studies examining the effects of light sources including irradiation distance and light-activation method on DC and PS are warranted in view of inconsistencies during clinical light curing and the various light polymerization regimens/modes available.

CONCLUSION

The DC and PS of contemporary bulk-fill RBCs including recently launched giomer materials were evaluated. Within the limitations of this study, the following conclusions can be drawn:

1. DC and PS of bulk-fill RBCs are product/depth dependent.
2. Bulk-fill RBCs should not be cured in more than 4-mm increments with the exception of SDR.
3. As DC of giomer bulk-fill RBCs at 4 mm depths was significantly lower than nongiomer bulk-fill materials, their clinical use at such depths should be exercised with caution.

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Conflict of Interest

The authors of this article certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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